Organic-free synthesis of layer-like FAU-type zeolites

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Supplementary Information

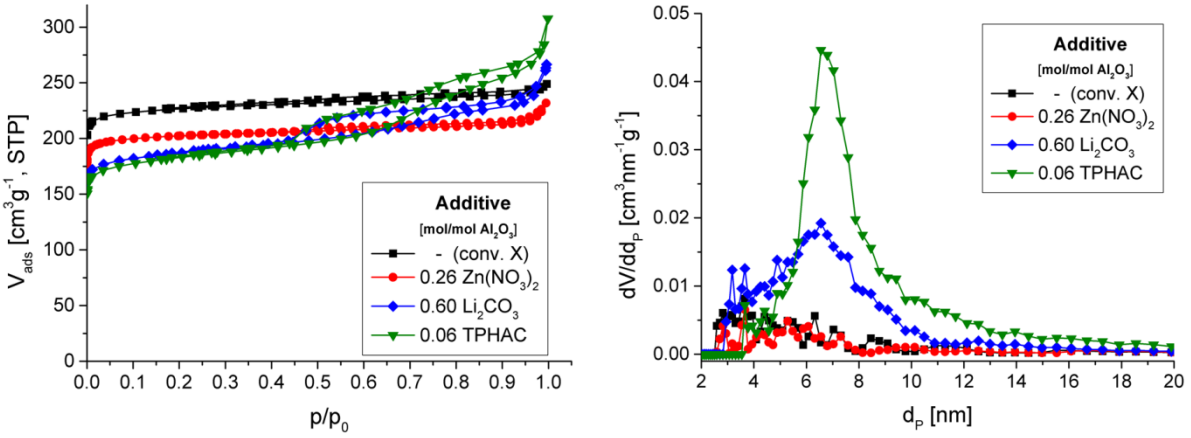

Fig. S1 Nitrogen physisorption isotherms and respective pore size distributions from the adsorption branch of the isotherm obtained with the NLDFT method for cylindrical zeolite pores

Table S1: Textural data from N_2 physisorption

Additive	A_{BET} [m^{2}g^{-1}]	V_{micro} [cm^{3}g^{-1}]	V_{meso} [cm^{3}g^{-1}]	V_{tot} [cm^{3}g^{-1}]
-	933	0.37	0.02	0.39
$Zn(NO_3)_2$	830	0.33	0.03	0.36
Li_2CO_3	748	0.29	0.12	0.41
TPHAC	723	0.27	0.21	0.48

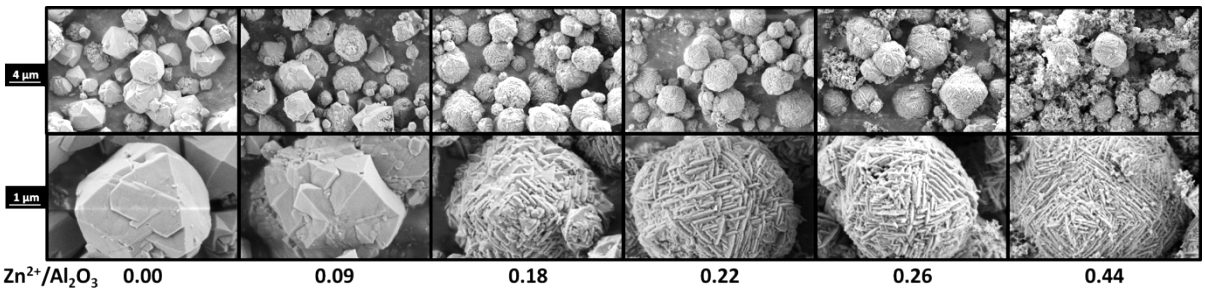

Fig. S2 SEM images of zeolite samples synthesised in the presence of increasing amounts of zinc nitrate, crystallization time: 15 days (at 75 °C); molar synthesis composition: 1 Al_2O_3 : 3.2 Na_2O : 3 SiO_2 : 150 H_2O : (0.00-0.44) Zn^{2+}

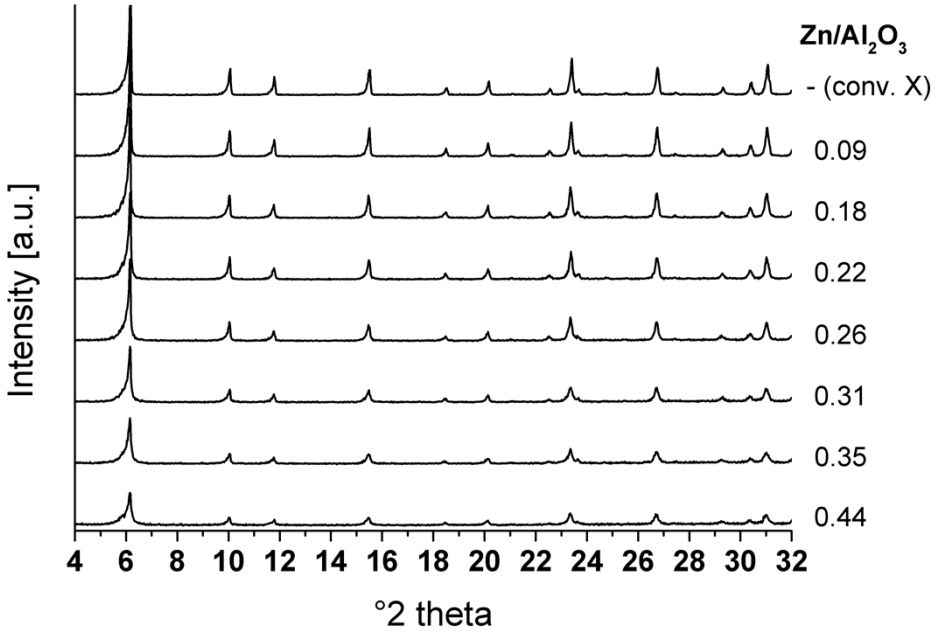

Fig. S3 XRD patterns of zeolite samples synthesised in the presence of increasing amounts of zinc nitrate; note that the crystallization time was only 15 days (at 75 °C)

Elemental analyses by ICP-OES (Table S2) showed Si/Al molar ratios of around 1.4 for the conventional zeolite X sample as well as for the one crystallized in the presence of zinc nitrate. Furthermore, with increasing amount of zinc nitrate in the synthesis gel increasing amounts of zinc were found in the sample, though in such small amounts that reliable conclusions on the degree of isomorphous substitution are not possible.

Table S2: Data from elemental analysis (ICP-OES, molar ratios) for zeolite samples obtained from zinc nitrate containing synthesis mixtures after 15 days of crystallization at 75 °C

Zn/Al set	Zn/Al	Zn [wt%]	Si/Al	Na/Al	(Na+Zn)/Al	(Si+Zn)/Al	Si/(Al+Zn)
0.00	0.00	0.00	1.43	0.98	0.98	1.43	1.43
0.09	0.02	0.57	1.44	0.98	1.00	1.46	1.41
0.18	0.04	1.13	1.40	0.95	0.99	1.45	1.34
0.22	0.06	1.71	1.36	1.03	1.09	1.42	1.29
*0.26**	*0.09*	*2.43*	*1.47*	*1.08*	*1.17*	*1.57*	*1.34*
*0.31**	*0.11*	*3.01*	*1.47*	*1.05*	*1.16*	*1.58*	*1.32*
*0.35**	*0.14*	*3.65*	*1.51*	*1.10*	*1.24*	*1.65*	*1.32*
*0.44**	*0.21*	*4.82*	*1.63*	*1.15*	*1.36*	*1.83*	*1.35*

*increasing amounts of amorphous material were present in these samples with increasing amount of zinc nitrate in the synthesis gel

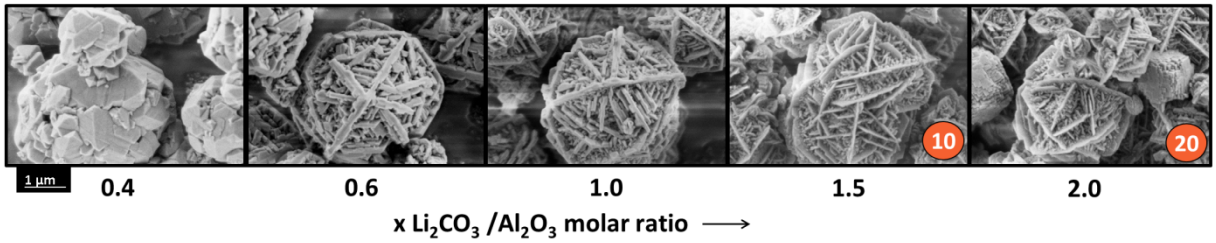

Fig. S4 SEM images of zeolite samples synthesised in the presence of increasing amounts of lithium carbonate, crystallization time: 28 hours (at 75 °C), number in the circles indicates the volume fraction of the by-products LTA and KFI-type zeolite ZK-5 (structural twin of zeolite LTA) observed in the SEM images; molar synthesis composition: 1 Al_2O_3 : 3.7 Na_2O : 3 SiO_2 : 180 H_2O : (0.00-0.60) Li^+

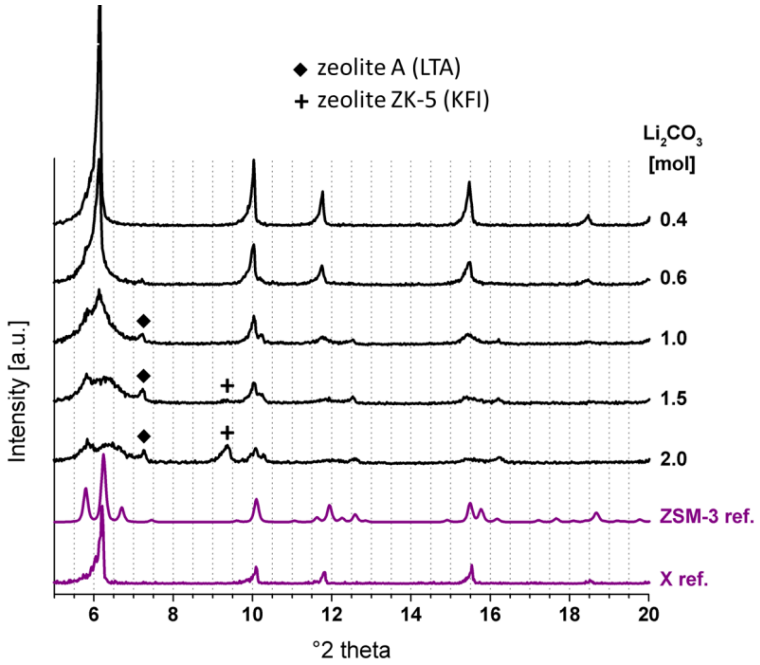

Fig. S5 XRD patterns of zeolite samples synthesised in the presence of different amounts of lithium carbonate (based on 1 mol Al_2O_3 in the synthesis composition), crystallization time: 28 hours (at 75 °C)

Experimental (analytics)

Powder XRD patterns were recorded in a Philips X'pert Pro diffractometer operated at 40 kV and 40 mA using CuK_α radiation in the °2 theta range from 2 to 50 in steps of 0.02 degree with a sampling time of 1 s per step. Before measurement the samples were stored for one day at ambient conditions and pestled into fine powder.

SEM pictures were recorded with the electron microscope ULTRA55 (Carl Zeiss MST AG). Samples were fixed on a conducting self-sticking carbon pad.

Nitrogen physisorption was conducted at 77 K in a Quantachrome Autosorb-1 instrument. Prior to the measurements the calcined samples were outgassed for 12 hours at 300 °C under vacuum. Pore size distribution curves were calculated from the adsorption branch of the isotherm using the Quantachrome DFT kernel for nitrogen at 77 K in cylindrical silica pores. Micropore volumes (V_{micro}) were determined as the DFT cumulative pore volume for pores below 2 nm pore diameter. The total

pore volume (V_{tot}) was calculated from the adsorption point at 0.99 p/p_0. Specific surface areas were obtained from the Brunauer Emmet Teller (BET) equation in the linear range between 0.01 and 0.20 p/p_0.

For elemental analysis inductively coupled plasma optical emission spectroscopy (ICP-OES, instrument Plasma 400, Perkin Elmer, USA) was used. Before measurement the samples were hydrothermally solubilized in a mixture of HF, HNO_3 and HCl during 90 minutes treatment in a microwave oven.